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AUTOMATIC CONTROL OF PARTICLE SIZE OF DIOCTYL PHTHALATE (DOP) A--ETC(U)
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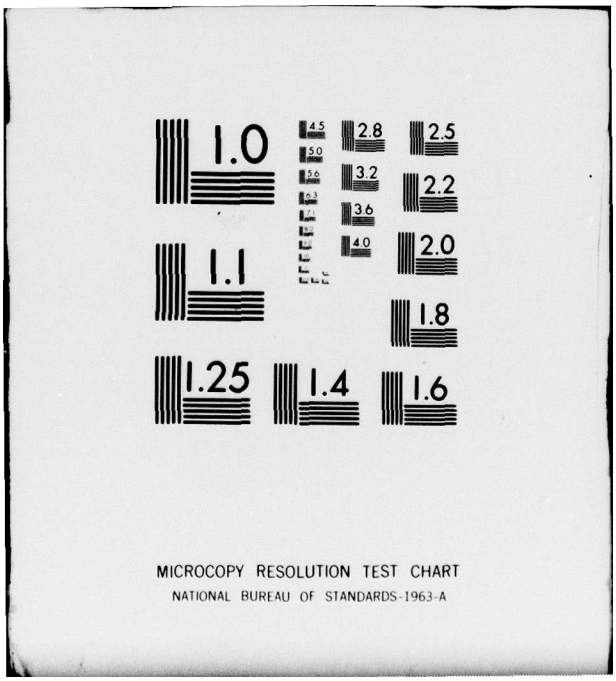
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6 AUTOMATIC CONTROL OF PARTICLE SIZE OF DIOCTYL PHTHALATE (DOP) AEROSOL USING A SIMPLIFIED PARTICLE-SIZE ANALYZER.

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ABSTRACT

The conventional particle-size analyser for the 0.3-μm dioctyl phthalate (DOP) aerosol used to test particulate filters was considerably modified. Under contract, a new particle-size instrument was developed which could be calibrated quickly and simply without the use of a calibrating lamp. Also, an output signal was generated for automatic control of particle size.

RÉSUMÉ

Des modifications majeures furent appatées à l'analyseur de taille de particules de type coorant utilisé dans les essais de contrôle de qualité des filtres à particules à l'aide d'un aérosol de phtalate de dioetyl (DOP) de 0.3 μm de diamètre.

Un contraetant développa un nouvel instrument d'analyse de la taille des particules d'un aérosol pouvant être calèbré facilement et rapidement sans l'utilisation d'une source lumineuse de calibration. Deplus, un système de rétroaction permettant un contrôle automatique de la grosseur des particules fut incorporé.

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AEROSOL GENERATION

High-efficiency particulate filters and filter media are tested with an aerosol of the di(2-ethylhexyl) ester of phthalic acid (dioctyl phthalate or DOP). Homogeneous aerosols of this material are produced by mixing flows of hot vapor and cold air so as to condense the DOP under controlled conditions (1). For laboratory evaluation, DREO has used a Model Q127 DOP penetrometer originally developed by the US Army but now manufactured by Air Techniques Inc., Baltimore, Md. This instrument produces about 100 1/min of smoke containing particles 0.3μm in diameter at a concentration of about 100 mg/m^3.

In the Q127 penetrometer, DOP vapor is generated by passing hot air over a reservoir of heated (172°C) dioctyl phthalate. To avoid having to heat a reservoir of liquid before being able to produce aerosol and make measurements and also to avoid any thermal degradation of DOP, a technique in which liquid DOP was injected directly into a stream of hot air was developed at DREO (2). For producing much larger quantities of smoke, liquid can be injected without special apparatus but because of the small rate (0.24 g/h) required for a laboratory instrument and because of the necessity for a steady flow, the liquid was sprayed from a hypodermic needle to a target by a 4000-V potential between them. An experimental penetrometer (DREO Serial No. 00-214) was built using this system and is described in an earlier report (2).

The size of aerosol particles condensed from DOP vapor is a function of the temperature of condensation. As shown in Figure 1 for the 00-214 instrument, flow rates for vapor and air for quenching were regulated with needle valves and measured with rotameters. Temperature of the vapor stream was automatically controlled with an electronic proportional controller. Condensation temperature and hence particle size were controlled by manually regulating power to the quench-air heater. The two flow rates and the temperature of the vapor were selected so that the temperature of the quench air producing the desired particle size was just sufficiently above ambient that it could be maintained with a low-wattage heater. This heater had a resistance of 7 ohms and a voltage supply from zero to about 12 V was sufficient to achieve the desired particle size of 0.3 μm. To ensure that the quench air was cool enough when the penetrometer was operated in extremely hot locations, a thermoelectric cooler was installed ahead of the quench-air heater.

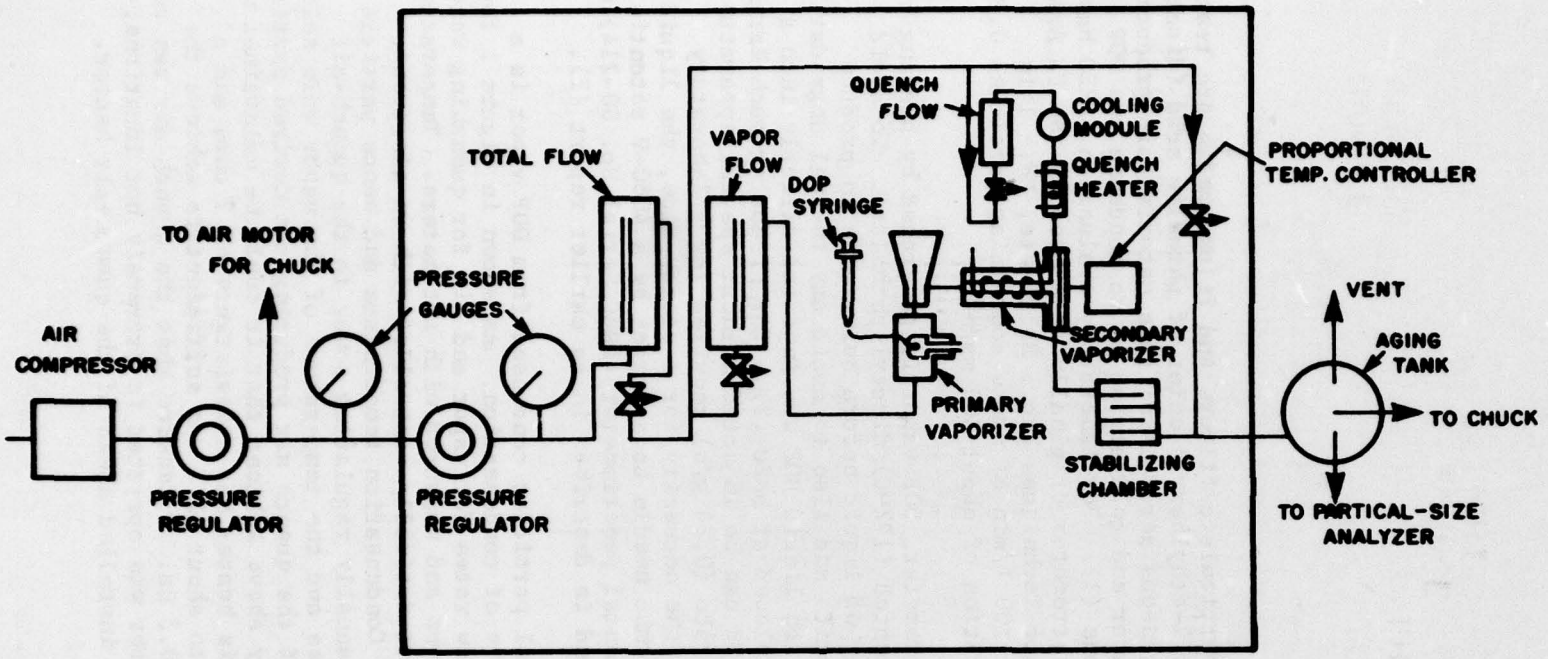

Figure 1. Flow diagram of smoke generator.

The smoke was further diluted with air at ambient temperature to obtain the desired concentration. This concentration was low enough to prevent significant coagulation of aerosol particles.

PARTICLE SIZE MEASUREMENT

The size of liquid aerosol particles in the range of 0.3 μm can be determined by measuring the degree of polarization of light scattered by the smoke (1,3). This can be done with either a visual or a photoelectric instrument (2,4). Only a brief description of the photoelectric method will be given here. A sample of the smoke (5 1/min) was passed through a chamber where it was illuminated with a parallel beam of white light. The light scattered at 90° to this beam at two opposite positions (see Figure 2) was viewed by two photomultiplier (PM) tubes. Between the smoke chamber and each PM tube were first a fixed Polaroid disc and then a rotatable disc. The fixed discs had their polarization axes at right angles to each other. The other two discs had their polarization axes parallel and were mounted so that they could be rotated together. At an angle setting of 29° between the common axis of these discs and the axis of one of the other discs the intensity of the light at the two PM tubes should be equal when the particle size is 0.3 μm (3,4).

Initially, vacuum-tube circuitry was used to compare electrical outputs of the two PM tubes. Later, solid-state designs were made available by Air Techniques Inc. With either type of circuitry it was necessary to match the gains of the PM tubes. This was done using a separate calibrating lamp to provide a source of unpolarized light. With no smoke in the chamber and with the scattering lamp off, the calibrating lamp was inserted into the chamber. With the analyzing Polaroids set at 45° the gains of the tubes were adjusted to zero the difference between them. To allow for non-linearity in response of the PM tubes, light output of the calibrating lamp had first to be adjusted to give the same intensity as the light scattered by the smoke. This was accomplished by setting the analyzer to θ° and adjusting the calibrating lamp intensity to make the output of one of the PM tubes the same as when smoke was being viewed with the analyzer at this same setting. With the analyzer at 0°, the PM tube having both Polaroids oriented with their axes in the same direction received the full output of the lamp while the other with discs oriented at right angles receiving no light.

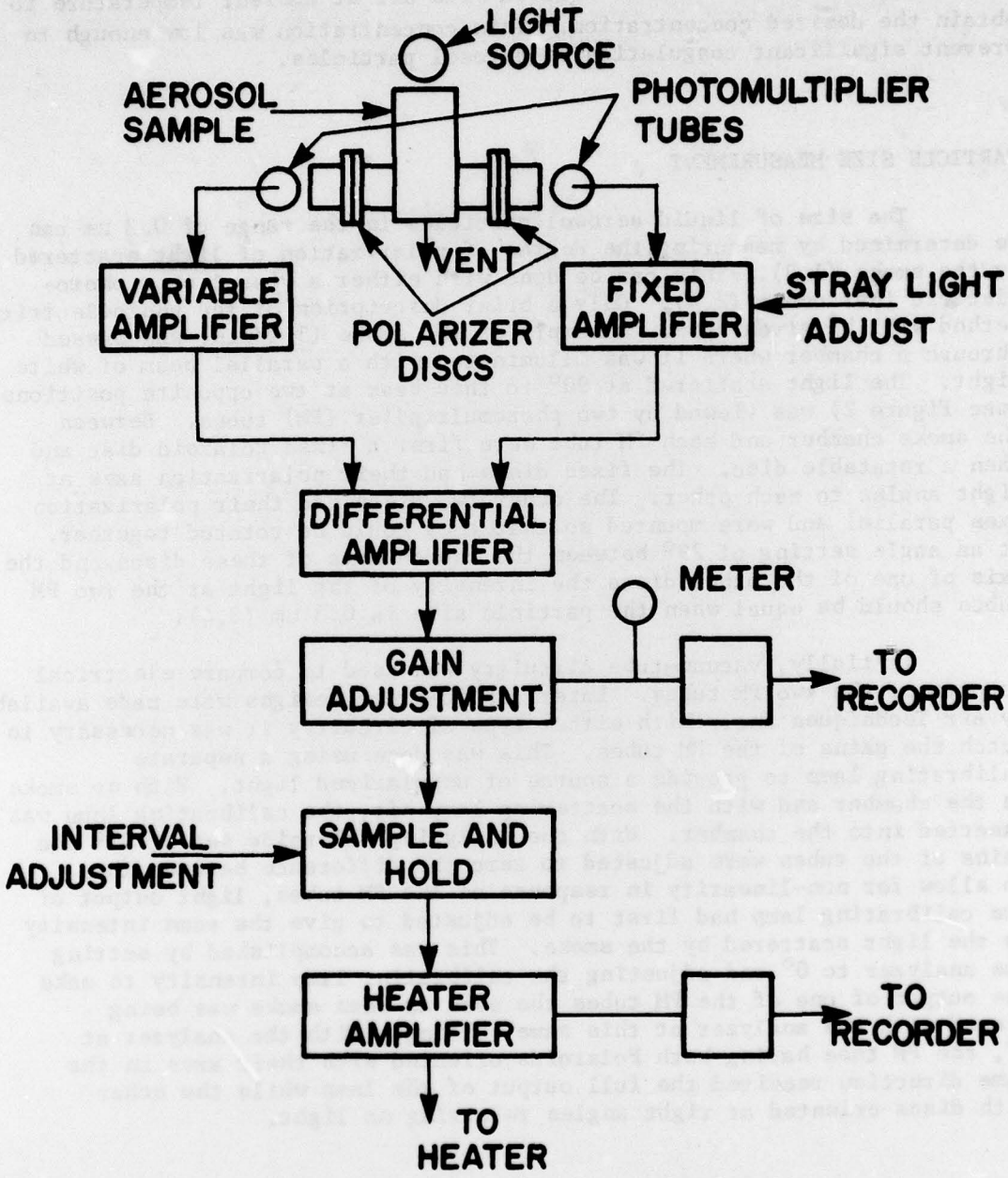

Figure 2. Block diagram of system for measurement and control of particle size.

The instrument was calibrated in such a manner that a rotation of the analyzer through 0.5° caused a known deflection of the error meter. Thus, during operation a change of particle size indicated by the meter could be directly related to polarizing angle. Testing specifications were that the angle be within one-half a degree of 29°.

The calibrating lamp consisted of an incandescent bulb mounted in a brass holder which fitted into the top of the smoke chamber. The holder had opal glass sides facing the two photomultiplier tubes. When not in use, the lamp sat in the top of the smoke chamber above the beam from the operating lamp and during calibration it was pushed down into the chamber.

The calibration procedure with this equipment was long and tedious particularly since some adjustments could not be made independently of the others. The use of a calibration lamp had several undesirable features.

- With the smoke chamber at negative pressure, air tended to leak in around the lamp holder.
- The orientation of the lamp filament with respect to the opal glass diffusers was very critical because the light intensity at the diffusers varied as the square of the distance between diffuser and lamp filament.
- The need to adjust the lamp to the same intensity as that scattered by the smoke greatly complicated the procedure.
- Three positions (0°,29°,45°) of the polarization analyzer had to be used during calibration.
- The glass diffusers collected liquid DOP causing unequal light on the two sides.

PSA-100 SYSTEM FOR MEASUREMENT

A contract was awarded to EDA Electronics Ltd., Ottawa, Ontario to develop a new system for measuring particle size. The main objectives were to obtain an instrument that was quick to warm up, stable during operation and simple to calibrate. As a second objective the possibility of automatic control of particle size was to be examined.

The contractor studied the existing system and decided to retain the same basic method for determination of particle size i.e. measurement of degree of polarization. Other methods of measuring the angle of polarization which avoided the necessity for optical/electrical gain calibration were considered but it appeared better to use the existing method with an improved calibration procedure in which no extra

calibrating lamp was used. The contractor also decided to continue to use photomultiplier tubes RCA No. 931A instead of other photo sensors.

The PSA-100 particle-size instrument made by EDA Electronics Ltd. and installed on the 00-214 penetrometer provided power for the 12-V light-scattering lamp and high voltage for the photomultiplier tubes. It amplified the output signals of the PM tubes and provided a meter reading and recorder output of the difference (Figure 2). The polarity and magnitude of this difference or error signal was an indication of particle size. The instrument was designed so that a 0.5-degree change in analyzer angle produced a 2-V change at the error meter. The gain of the amplifier for one tube was fixed but the other was provided with a gain adjustment to enable the responses to be matched. Also, the error signal between the two tubes was used to generate a feedback to the quench-air heater to automatically correct for particle-size error. This will be described in the next section of the report.

To dispense with the calibrating lamp in the PSA-100 particle-size instrument, the fixed polarizer in one of the side arms of the analyzer was remounted so that for calibration purposes it could be rotated through a fixed angle of $90^{\circ} \pm 0.05^{\circ}$. When it was in the rotated position the optics of the two arms were identical and the outputs of the two photomultiplier tubes could be matched using the polarized light scattered from the smoke. Although matching could be done at any angle of the analyzer except near 0° where all light would be blocked, it was most convenient to leave it at the 29° setting used for 0.3-μm particles. This assured that the light intensity at which the tubes were matched was the same as that during operation. With this simplified procedure the calibration could be checked at any time merely by rotating the polarizing disc to the calibrate position.

The validity of using this method was verified by balancing the PM outputs using the calibrating lamp with the analyzer at 45° and then switching to the new calibrating orientation (analyzer at 29° and polarizer rotated 90°). There was no change in the balance.

A lamp voltage control was incorporated into the PSA-100 instrument. The concept was that if the smoke concentration changed, the photo tube gains might no longer be equal. The output of one tube would then be used to vary the lamp voltage to restore the light intensity to the previous level. This might have been effective if particle-size changes did not cause changes in the intensity seen by each photo tube. The changed polarization due to the change in particle size would result in an increased light intensity at one photo tube and a decreased intensity at the other. The lamp control circuit therefore was disconnected.

No means was provided in the PSA-100 to adjust for stray light signals from the PM tubes or to compensate for difference in dark currents. A facility was built into the input to the fixed amplifier to impose a variable voltage either positive or negative to null the error meter under conditions of no smoke.

AUTOMATIC CONTROL OF PARTICLE SIZE

It is evident that once an electrical signal related to particle size and a means of regulating the size were available a device could be installed to close the loop and achieve automatic control. In order to save the cost and space of a separate controller EDA Electronics Ltd. was requested to incorporate the control function into the particle-size instrument. This was done by providing a 0-to-12-V dc output for driving the quench-air heater with about 50% of the output to the heater corresponding to zero error signal.

Because of the time lag between smoke generation and measurement of particle size a sample-and-hold system of measurement and control was used. The instrument took a one-second sample of the particle-size error during which time the heater voltage was adjusted according to the error. This adjusted voltage was held until the next sampling period. The sampling interval could be varied from 1 to 99 s by means of a two-digit thumbwheel switch on the front panel of the instrument.

In order to lessen the time lag as much as possible, the sample of aerosol for particle-size measurement was taken before the aging tank instead of after as shown in Figure 1. The chamber had a capacity of 4.5 l which at 50 l/m flow rate gave an average retention time of 5.4 s.

Figure 2 is a block diagram of the circuitry used to measure and control particle size. As initially supplied, the PSA-100 instrument had a switch to give a gain of either 10 or 1 to the error signal with an additional gain-adjustment potentiometer on the front panel. This High/Low switch was found to be unnecessary as the potentiometer allowed adequate adjustment with the switch at the high position.

Since no adjustment was provided for the heater amplifier, proportional band or sensitivity of the control process had to be adjusted by changing the error gain. When this was done the relationship between chart reading and polarizer angle (particle size) was altered. After one modification which was only partially successful, a two-deck,

ten-position rotary switch was installed. On one deck nine 100-kΩ resistors were mounted between terminals to give 0 to 0.9 MΩ in ten steps. This was connected to replace the error-gain potentiometer. The other deck had nine 1-kΩ resistors and was used with a 1.2-kΩ resistor to give a voltage divider which varied the meter and recorder signal in a manner inversely proportional to the error gain. This enabled the error-gain adjustment to be changed without significantly changing the particle-size error reading.

As modified, the instrument gave a control output of 7 V for an error signal of zero which, across a 7-Ω heater, was a power of 7 watts. Over the working range, heater power varied 0.95 watts per volt change in the error.

EXPERIMENTAL

The aerosol generator was started with the analyzer set at 29° and the error gain at the lowest sensitivity. With the temperature and flow of the vapor stream constant, the flow of quench air was adjusted to give an error signal of zero and thus a particle size of 0.3 μm. Control was then initiated by increasing the gain setting until hunting began to be noticeable. The optimum setting of the sampling interval was about 2-3 s.

As an evaluation of particle-size control, a variable dc power supply was connected to the quench-air heater so that power to the heater could be alternately switched from manual to automatic. When left on manual control without any change in the heater power there was a long-term drift of particle size which followed changes of ambient temperature and build-up of heat in the compressed-air tank of the aerosol generator (2). Superimposed on this long-term drift was a regular cyclic change of particle size. The cycle repeated with each revolution of the screw of the DOP infusion pump indicating small changes in rate of DOP injection and consequent changes of vapor temperature which were not immediately corrected by the temperature controller. When on automatic control, the long-term drift became negligible and the cyclic variations were considerably reduced. The combined variations were within acceptable limits i.e., not more than caused by moving the analyzer ±0.5° from the 29° setting.

To determine how much temperature change the automatic control could compensate for, the quench-air flow was alternately passed through the quench-air cooler and by-passed around it. Passing through the cooler reduced the temperature of the air entering the quench-air heater by more than 20°C. The change in error signal, after the heater power had stabilized, was within the acceptable limits mentioned above.

Figure 3 shows the variation in particle size and the resultant change in voltage to the quench-air heater. Particle size of course is shown in terms of analyzer angle. Chart reading in terms of angle was determined by rotating the analyzer to 28° then to 30°. The limits of ±0.5° were then marked at one-half of the deflections obtained.

CONCLUSIONS

The PSA-100 instrument greatly simplified the complex procedure for calibrating the photomultiplier tubes of the particle-size analyzer. The calibration can be checked and if necessary, corrected in a few seconds. The automatic control capability eliminated the necessity for constant monitoring of the particle size by the operator. Particle size was automatically maintained despite changes such as ambient temperature, which would otherwise affect particle size.

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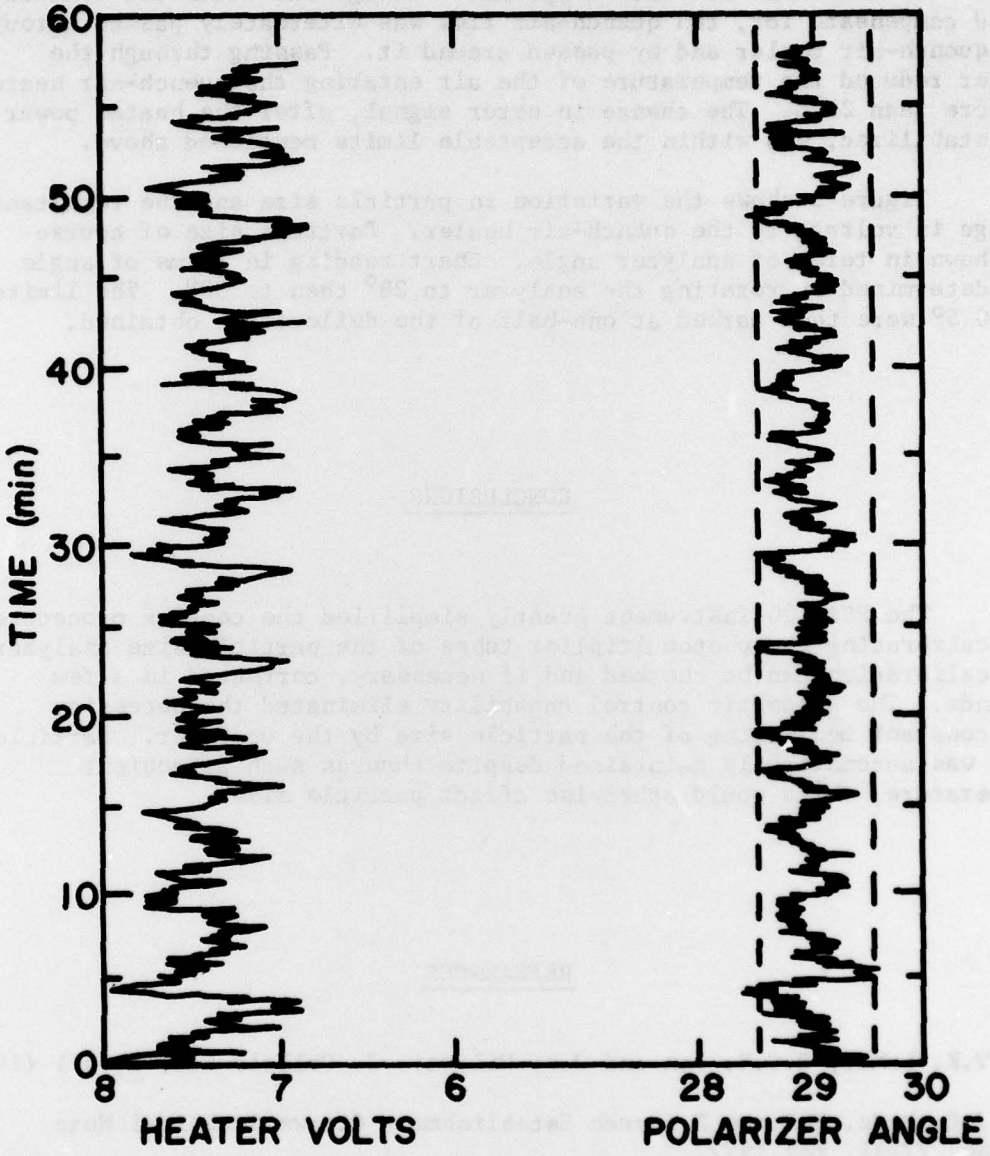

Figure 3. Portion of recorder chart showing fluctuations in particle size and output to quench-air heater.

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